

**A STUDY OF THE DEGRADATION OF DACRON FABRIC
BY THE ACTION OF CERTAIN CHEMICALS**

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A STUDY OF THE DEGRADATION OF DACRON FABRIC
BY THE ACTION OF CERTAIN CHEMICALS

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By
Albert Talmadge Taylor, Jr.

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TABLE OF CONTENTS

	Page
ACKNOWLEDGMENTS	ii
LIST OF TABLES	v
LIST OF ILLUSTRATIONS	vi
SUMMARY	viii
CHAPTER	
I. INTRODUCTION	1
Historical	
The Dacron Polymer	
Purpose of this Investigation	
II. THEORETICAL CONSIDERATIONS	9
Polymer Fibers	
Character of Dacron	
The Action of Carriers	
III. INSTRUMENTATION AND EQUIPMENT	15
IV. PROCEDURE	17
Sample Preparation	
Testing Procedure in General	
Tests at Room Temperature	
Tests at 120°F. and 160°F.	
Tests at the Boiling Point	
Sample Removal and Drying	
Surface Examination	
Changes in Hand and Coloration	
Shrinkage	
Breaking Strength and Elasticity	
V. DISCUSSION OF RESULTS	22
General	
Tensile Strength	
Elasticity	
Shrinkage	
Surface Characteristics	

	Page
VI. CONCLUSIONS	44
VII. RECOMMENDATIONS	46
BIBLIOGRAPHY	47
APPENDIX	51

LIST OF TABLES

Table	Page
1. Exposure in Tap Water	52
2. Exposure in 2 Per Cent Benzoic Acid	53
3. Exposure in 4 Per Cent Benzoic Acid	54
4. Exposure in 6 Per Cent Benzoic Acid	55
5. Exposure in 2 Per Cent Salicylic Acid	56
6. Exposure in 4 Per Cent Salicylic Acid	57
7. Exposure in 6 Per Cent Salicylic Acid	58
8. Exposure in 2 Per Cent Phenol	59
9. Exposure in 4 Per Cent Phenol	60
10. Exposure in 6 Per Cent Phenol	61
11. Exposure in 2 Per Cent Sodium Hypochlorite	62
12. Exposure in 4 Per Cent Sodium Hypochlorite	63
13. Exposure in 6 Per Cent Sodium Hypochlorite	64
14. Exposure in 2 Per Cent Monochlorobenzene	65
15. Exposure in 4 Per Cent Monochlorobenzene	66
16. Exposure in 6 Per Cent Monochlorobenzene	67
17. Exposure in 2 Per Cent <u>Ortho</u> -Phenyl Phenol	68
18. Exposure in 4 Per Cent <u>Ortho</u> -Phenyl Phenol	69
19. Exposure in 6 Per Cent <u>Ortho</u> -Phenyl Phenol	70

LIST OF ILLUSTRATIONS

Figure		Page
1.	Preparation of Dacron (Polyethylene Terephthalate)	4
2.	Effect of Time on Breaking Strength of Dacron Treated with 6 Per Cent Phenol at Various Temperatures	23
3.	Effect of Time on the Strength of Dacron in the 6 Per Cent Concentration Series at 210°F. . .	24
4.	Effect of Time on the Elasticity of Dacron Treated in the 6 Per Cent Concentration Series at 210°F.	27
5.	Effect of Time on the Elasticity of Dacron Treated in the 4 Per Cent Concentration Series at 210°F.	28
6.	Effect of Time on the Elasticity of Dacron Treated in the 2 Per Cent Concentration Series at 210°F.	29
7.	Effect of Time on the Elasticity of Dacron Treated in the 6 Per Cent Concentration Series at 160°F.	30
8.	Effect of Time on the Elasticity of Dacron Treated in the 4 Per Cent Concentration Series at 160°F.	31
9.	Effect of Time on the Elasticity of Dacron Treated in the 2 Per Cent Concentration Series at 160°F.	32
10.	Effect of Time on the Elasticity of Dacron Treated in the 6 Per Cent Concentration Series at 70°F.	33
11.	Effect of Time on Shrinkage of Dacron Treated in the 6 Per Cent Concentration Series at 210°F. .	36
12.	Effect of Time on Shrinkage of Dacron Treated in the 4 Per Cent Concentration Series at 210°F. .	37
13.	Effect of Time on Shrinkage of Dacron Treated in the 2 Per Cent Concentration Series at 210°F. .	38

Figure	Page
14. Effect of Time on Shrinkage of Dacron Treated in the 6 Per Cent Concentration Series at 160°F. .	39
15. Effect of Time on Shrinkage of Dacron Treated in the 4 Per Cent Concentration Series at 160°F. .	40
16. Effect of Time on Shrinkage of Dacron Treated in the 2 Per Cent Concentration Series at 160°F. .	41
17. Effect of Time on Shrinkage of Dacron Treated with 6 Per Cent Phenol at 70°F.	42

A STUDY OF THE DEGRADATION OF DACRON FABRIC
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SUMMARY

The success experienced in applying dyes to Dacron in the absence of pressurized equipment depends on the use of certain chemicals classed as swelling agents or carriers, while finishing processes and subsequent end use of Dacron fabric require the use of bleaching agents. The effects of these agents on certain fiber characteristics have not been extensively investigated, therefore many questions have been raised, such as: What is the effect on tensile strength? What is the effect on shrinkage? What is the effect on elasticity? What is the effect on surface characteristics of the fiber?

The purpose of this investigation was to answer these questions for a limited group of chemical agents and thereby increase the empirical knowledge available to the dyeing industry. In this case it was considered that even negative information would be of value if only by virtue of the fact that these questions must be answered if the field of carrier dyeing is to be explored thoroughly.

Studies were made on the influence of three variables: concentration, temperature, and time. The

chemical agents used were: benzoic acid, monochlorobenzene, ortho-phenyl phenol (sodium salt), phenol, salicylic acid, and sodium hypochlorite.

The concentration series of 2 per cent, 4 per cent, and 6 per cent was selected since it covered both the minimum and maximum amounts of these agents that might be employed in textile processing. Preliminary screening tests were conducted as an aid in selecting conditions that gave some indication of fiber degradation.

The influence of temperature was studied at the boiling point of the solution of the chemicals (210°F.), 160°F., 120°F., and room temperature. This selection was considered broad enough to give degradation within the time limit chosen.

The range of time intervals chosen for treatment varied from one hour to four weeks. Four different time intervals were used for each temperature, the longer time intervals being used at the lower temperatures.

Tests at the boiling points of the solutions of the chemical agents were made under reflux; for tests at 120°F. and 160°F., an electrically heated, thermostat-controlled waterbath was employed. Tests at room temperature were carried out in a room where temperature and relative humidity remained constant at 70°F. and 65 per cent RH respectively.

Results of this study indicated that tensile strength is unaffected by benzoic acid, monochlorobenzene, phenol,

salicylic acid and sodium hypochlorite. The sodium salt of ortho-phenyl phenol in 4 and 6 per cent concentrations, at the boil, caused strength losses up to the point of complete degradation; however, there was no evidence of strength losses at other temperatures. Increases in shrinkage and gains in elasticity were greatest with the phenols and monochlorobenzene. Some shrinkage may be attributed to fabric geometry, while increased swelling is considered partially responsible where greater percentages of shrinkage were evidenced. Elasticity did not prove to be a function of shrinkage as might have been expected. The magnitude of the elasticity gains for fabric treated with phenols and monochlorobenzene indicated that the molecular structure may have been affected. Changes in fabric hand and coloration were most prevalent in the 4 and 6 per cent concentration series of the phenols and monochlorobenzene.

No attempt was made to draw conclusions from the results of this investigation as to the nature of the chemical action of the different agents used or of the possible rearrangement of the fiber structure.

CHAPTER I

INTRODUCTION

Historical.--The recent introduction of Dacron polyester fiber is one illustration of the dynamic nature of the textile industry. For example, in 1800 the fiber used predominantly for clothing was wool. Now, if we jump to 1950 we note a great change in the fibers used for wearing apparel in the United States. Fiber consumption for wearing apparel in the order of decreasing consumption is as follows: cotton, rayon, wool, nylon, silk and other allied fibers. The basic change in the textile industry during this 150 year span occurred with the introduction of rayon, nylon, acrylic fibers, Vicara, and Dacron polyester fiber. With the advent of the newer man-made fibers came many problems; outstanding were those in the field of dyeing techniques.

Man's first attempts to duplicate the beauty and color of his environment are lost in antiquity. His early efforts probably consisted of staining his crudely woven cloths with colored juices, obtained from flowers, fruits, and the bark of trees. The variety of shades was limited, and for the most part did not survive exposure to sunlight and washing.

Prehistoric efforts at dyeing fabrics and objects were

hampered by the fact that few natural dyes formed fast combinations with the materials dyed. But it was found eventually that this lack of affinity could be overcome by mordants, substances which cause the dye to adhere firmly to the fabric. It was not until this discovery was made, probably in India before 2000 B.C., that dyeing made any appreciable progress.

The modern techniques of dyeing some of the man-made fibers with carriers or swelling agents which swell and supposedly carry the dye into a hydrophobic fiber may be likened in some respects to the discovery of mordants. Without these agents the production of dyed fabric from some fibers would be extremely limited. It is with the action of these agents that this investigation is primarily concerned.

The groundwork for the development of Dacron was laid by the late Dr. W. H. Carothers, duPont research chemist, in his work on high polymers. Polyesters were among the first condensation polymers investigated by Carothers; however, instead of continuing with an exhaustive study of polyesters, Carothers elected to devote the major portion of his efforts to polyamides, a course which resulted in the development of nylon (1).

British research chemists of the Calico Printers Association Ltd., after studying the published works of Dr. Carothers, initiated a further study of polyesters. Their work led to the development of a polyester fiber

now known in England as Terylene. In 1946 DuPont purchased the patent rights to this polyester fiber under the provisional title of "Fiber V" and since that time an intensive development program has been in effect. The first commercial production of Dacron took place in 1953 at the DuPont plant located in Kinston, North Carolina (1) (2).

In chemical terms Dacron is polyethylene terephthalate, a linear polymeric fiber, obtained by the condensation of terephthalic acid with ethylene glycol. Dimethyl terephthalate, a flaky white powder made from nitric acid, xylene and methanol is the principal raw material for the fiber. Commercial preparation involves the following steps: (Figure 1)

1. Direct esterification of terephthalic acid.
2. Catalyzed ester interchange between the resulting dimethyl terephthalate and excess ethylene glycol (3).

The resulting condensate is heated in vacuo to effect removal of glycol and give polyethylene terephthalate, a product of molecular weight between 8000 and 24,000 (3) (4). Polyethylene terephthalate is amorphous as it is cast from the reaction vessel and retains the amorphous form on rapid cooling. Crystallization occurs on heating, and the polymer loses its transparent glassy appearance, becoming pale cream in color.

The polymer is converted to filaments by the melt extrusion method. In this step, it is essential that all

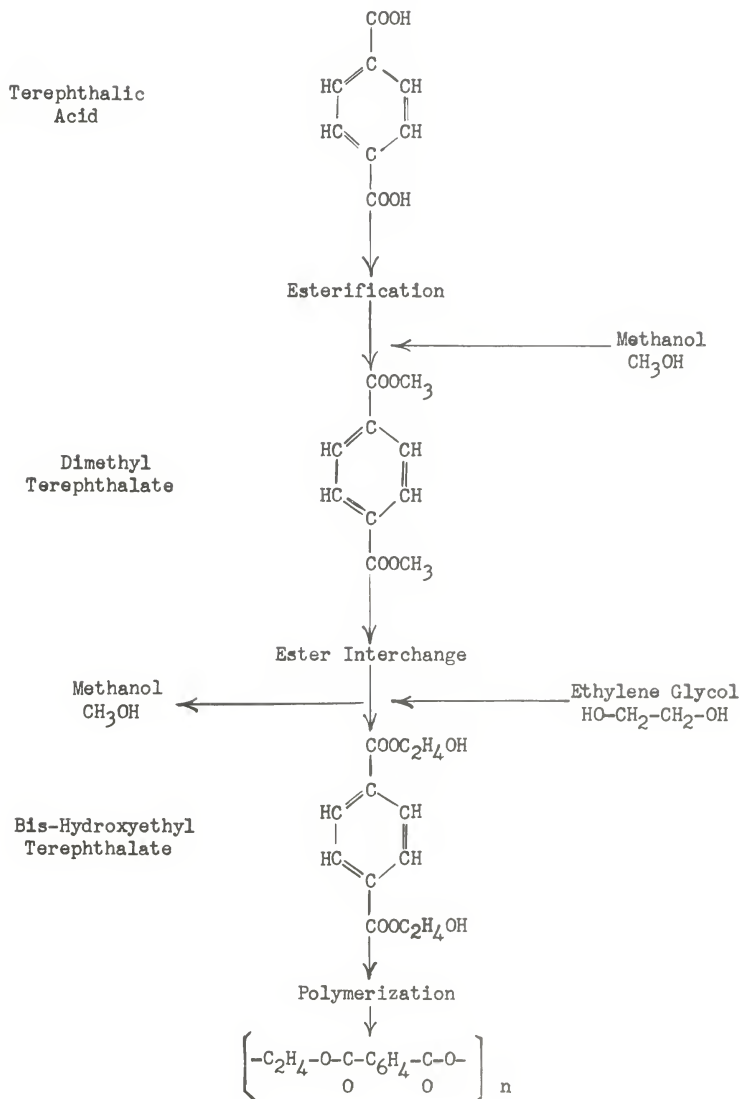


Figure 1. Preparation of Dacron (Polyethylene Terephthalate)

traces of water be removed, prior to fusion, as otherwise hydrolysis and immediate decrease in molecular weight will result (5).

The Dacron Polymer.--Dacron has good resistance to most weak acids at boiling temperatures, and to strong acids at room temperature. It is disintegrated by concentrated (96 per cent) sulfuric acid. The fiber has good resistance to strong alkalis at room temperatures, but is degraded by strong alkalis at elevated temperatures. Dacron has excellent resistance to oxidizing agents and is not degraded by bleaching treatments normally used for other textile fibers (6).

In the attempt to make fibers of outstanding tensile strength, dimensional stability and chemical resistance, highly crystalline polymers such as Dacron are subjected to stretching and heat setting treatments to yield dense, impermeable and very strong fibers into which no dye molecule can penetrate (7). Due to the inertness of the fiber and its compact structure, Dacron was said to be undyeable a few years ago (8).

Dyeing processes in the textile industry were developed for hydrophilic fibers whereas almost all of the newer man-made fibers are hydrophobic in nature; this brought into use the dispersed acetate dyes which were developed primarily for the hydrophobic cellulose acetate fiber. Polyester fibers have an affinity for these dyes but are much more resistant to dyeing than cellulose

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acetate. The chemical similarity between Dacron and cellulose acetate is clearly shown by the fact that ester groups constitute 41 per cent by weight of acetate and 46 per cent by weight of Dacron (9).

Since the dispersed acetate dyes show poor penetration and exhaustion even at the boil on Dacron, experiments were carried out to determine whether this was due to inability to absorb dye or due to difficulty of the dye to diffuse into the fiber. Results of these experiments showed the capacity to sorb was as great as cellulose acetate and that the fiber was even more dyeable than nylon; however at 185°F. these dyes diffuse 500 times faster in cellulose acetate and 700 times faster in nylon than in Dacron (10).

Further experiments were conducted in which Dacron was allowed to dissolve slowly in caustic soda and it was observed that as the filaments slowly decreased in diameter the relative rates of diffusion did not change. The effect is not due, therefore, to a resistant skin on the fibers but arises from the hydrophobic character and compact physical structure of the fiber as a whole. Further, the dispersed acetate dyes have a great affinity for ester sites and with the number of ester groups present in Dacron the poor dyeability cannot be attributed to the lack of dyeing sites (11).

Experimental data thus clearly emphasized the need for a method to increase the rate of diffusion of dyes into

the fiber. Three possible lines of approach were as follows:

1. The use of dyes of smaller molecular size.
2. The use of swelling agents or carriers to increase the permeability of the fiber.
3. The use of pressure at elevated temperatures (above 212°F.).

All three of these methods have been used with varying degrees of success; however, the field of carrier dyeing is the most widely used at present (12).

Purpose of this Investigation.--The purpose of this study was to determine the degrading effect, if any, that a limited group of swelling agents and one oxidizing agent would have on Dacron fabric. Six chemicals commonly used in dyeing and finishing of Dacron were employed for various times, at various temperatures and concentrations in order to bring about the degradation. Studies were made to determine the effects on tensile strength, elasticity, shrinkage and surface characteristics.

Preliminary screening tests were performed on the fabric to determine possible changes or trends in strength, elasticity and shrinkage produced by the agents chosen. The temperature and time ranges were set to cover as broad a scope as possible over which chemical action would take place and over which the fabric might be used in combination with carriers.

From these studies it was felt that empirical information could be supplied on the degrading action of the six chemicals as a function of three variables: concentration, temperature, and time.

CHAPTER II

THEORETICAL CONSIDERATIONS

Polymer Fibers.--The properties of polymer fibers are dependent upon the extent and nature of the crystallinity which is possible for a given macromolecular structure in the quenched, annealed or drawn condition. Polymer fibers are heterogenous in nature consisting of crystalline and amorphous regions; transition from one to the other being gradual. Crystallinity is dependent on chemical structure which in turn influences intermolecular forces. These forces consist of hydrogen bonding systems, strongly polar groups and readily ionizable segments all of which form points of inter-chain attraction which can result in crystallinity. In general, the chemical nature of the chain-forming compounds accounts for the chemical properties of the polymer, while the inter-molecular forces are responsible for its physical characteristics (13).

The inter-molecular forces come into action only at very short distances. If the polymer chains fit together in such a manner that many atoms of one are close to the atoms of the other, the total molecular attractive energy will be large. If the degree of packing is high enough, the polymer will tend to crystallize. Thus the attractive

forces between linear molecules, which constitute the man-made fibers, are much greater if the molecules are orderly arranged in crystallites than if they are in an amorphous condition (14, 15, 16).

Character of Dacron.--No reliable information has been published on the molecular weight of Dacron since its limited solubility and high melting point present considerable difficulties in obtaining dependable values by such methods as viscosity, osmotic pressure and determination of end groups.

The crystalline structure of Dacron has been shown to be nearly planar in character, and the arrangement of the molecules can be regarded as the result of close packing, taking into account the normal Van der Waals radii of the atoms where the projections of one molecule fit into the hollows of the next (17).

In drawn polyethylene terephthalate the crystalline regions are usually very precisely oriented, though not quite parallel to the fiber axis, the angle of tilt being about 5° . No conclusions on orientation in the amorphous regions can be drawn from X-ray diffraction patterns, for drawn fibers show no "amorphous band" (18).

The Action of Carriers.--The theory of carrier dyeing is well summed up in the following statements by Vickerstaff (19).

The mechanism by which such agents promote dyeing is not understood. One theory is expressed in the name "carriers," which is often applied to these compounds, by which it is suggested that the substances form a complex with the dye molecule and "carry" the dye into the fiber for which the assistant has a greater affinity than the dye. It is difficult to visualize this process since it is unlikely that the complex of two molecules could diffuse more rapidly than either component alone. An alternative possibility is that the agents may function as molecular lubricants, being adsorbed on the fiber molecules and so reducing the inter-molecular cross-linking forces and pushing the fiber molecules apart by mere mechanical action. The greater freedom of motion of the polymer chains so produced and the disturbance of the crystalline structure will facilitate diffusion into the fiber by reducing the activation energy needed for the dye molecule to force its way through the entangled chains. Such a mechanism need not involve swelling or increased water uptake by the fibers. It is noteworthy that all effective dyeing assistants do produce either swelling or length shrinkage of the filaments or both, and the use of excessive quantities must be avoided if the physical properties are to be maintained.

An outstanding feature of Dacron and other hydrophobic fibers is the high negative zeta potential which they develop in aqueous solution. Although no measurements have been made on Dacron it has been found that the fiber reacts with anions only with extreme difficulty. It seems that an electrical potential barrier of such magnitude is set up at the fiber surface that the approach of any negative ion under normal conditions is prevented. For this reason anionic dyes or dyeing assistants are unable to enter the fibers (20).

In dyeing with dispersed acetate dyes it will be noted that very few contain carboxylic or sulfonic groups; it has been found that hydroxyl groups either in the side

chain or in the ring structure plus oxygen atoms, especially ketonic oxygen, such as is found in the 9, 10 positions in anthraquinone make for better dyeability with dispersed acetate colors. Phenolic hydroxyl groupings in dyes work as built-in swelling agents and the action is greatly accentuated when the hydroxyl group is in the para position. Free amine groupings possess certain positive dye value because of the basic nature and attraction for hydrophobic fibers. Ethylenic groups, hydrophobic in nature, also exert a positive influence on the dye uptake of Dacron (21).

The action of carriers on Dacron may be likened unto the action of water on viscose rayon. In the case of viscose in the unswollen state the fiber is highly impermeable to large dye molecules; however, in an aqueous dyebath the fiber swells, due to its hydrophilic nature, and thus increases the permeability of the fiber to dyes. The ionized groups in the fiber contribute to the effect through osmotic action, while the water breaks the hydrogen bonds cross-linking the fiber molecules, thus reducing the rigidity of the molecular network and allowing the dye molecule to enter the substrate. In hydrophobic fibers no such action takes place; however, certain compounds may be expected to produce a similar effect. Phenol proves to be the simplest example in the case of Dacron as it is absorbed by hydrogen bonding and in turn attracts water into

the fiber thus leading to swelling action. Phenol, being strongly hydrophilic in nature has been shown to give a marked increase in the rate of diffusion of cellulose acetate dyes into Dacron. Although this explanation of carrier action is plausible, the examination of thousands of compounds in the search for better carriers has shown many highly effective agents which could not possibly function in this manner (22).

Although the selection of a carrier remains largely empirical, the following categories seem to account for the greater number: (23)

1. Aromatic carboxylic acids.
2. Phenyl phenols.
3. Chlorinated benzenes.

In the case of hydrophilic carriers it is thought that the molecular lubrication mechanism may be operative; however, the distribution of the carrier between the fiber and the aqueous dyebath seems to be the important factor in determining the carrier efficiency. This accounts for the fact that compounds containing ionizable or solubilizing groups are notably ineffective; it has been shown that benzoic acid is much inferior to benzene and beta-naphtholsulfonic acid to beta-naphthol. Normally an increase in molecular weight of the agent, produced by non-solubilizing groups, serves to reduce the water solubility and increase absorption; however, this process may be pushed

too far and the molecule of the agent becomes too large to diffuse into the fiber. This is illustrated in the case of ortho-chlorophenol which is more effective than phenol but pentachlorophenol is less effective than ortho-chlorophenol (11).

The foregoing discussion of literature surveyed serves to point out the fact that little is known of carrier action or mechanism in the field of dyeing.

CHAPTER III

MATERIALS AND EQUIPMENT

The fabric used was filament Dacron of the following specifications:

Weight, oz/yd ²	1.9
Thickness, inch, (maximum)	0.0039
Breaking strength, pounds, (ravel strip test, 1 x 3 inch sample)	
Warp	80.8
Filling	50.2
Weave	Plain
Dimensions	
Width, inches	45
Length, yards	30
Construction	
Ends/inch	112
Picks/inch	72
Count	
Warp, denier	70 (34 filament)
Filling	70 (34 filament)

All instruments and machinery used were standard testing equipment found in the laboratories of the A. French Textile School.

Erlenmeyer flasks of 500 ml. capacity and 16 inch reflux condensers were used for tests at the boil. For tests conducted at 160°F. and 120°F., 500 ml. porcelain beakers were used.

An electrically heated, thermostat-controlled waterbath was used for all tests at 120°F. and 160°F. The waterbath was heated by a 5 KW. immersion type heater and the temperature was held constant within plus or minus 2°F. The waterbath was constructed of galvanized steel and was of the following dimensions: 35 inches long, 20 inches wide, 6 inches deep. The cover of the bath contained thirty-two 3 1/4 inch diameter holes which held the porcelain beakers. Standard watch glasses were used to cover the beakers and holes when not in use.

A Beckman Model H-2 Glass Electrode pH Meter, equipped with a standard calomel electrode and a glass electrode with a temperature range from 15°C. to 100°C., was used to measure the pH of all solutions.

A Scott J-2 Vertical Tester with 2 inch jaws was used for breaking strength tests.

A standard student type microscope was used in examination of surface characteristics.

CHAPTER IV

PROCEDURE

Sample Preparation.--Strips 6 1/2 inches wide were cut filling-wise from the Dacron fabric; each strip was then cut into 9 inch lengths. The 6 1/2 x 9 inch samples were treated, and later tested for shrinkage, examined microscopically for surface changes, examined for changes in hand and color and finally for tensile strength and elasticity. For tensile strength tests each sample provided five 6 x 1 1/2 inch specimens. Specimens were ravelled down to 1 inch in width in accordance with the ravelled strip testing procedure for tensile strength. Warp breaking strength only was tested in this investigation.

In order that treated samples might be compared with the original untreated fabric for tensile strength a method was devised whereby the same number of warp ends per specimen would be tested. This was accomplished by marking off the 6 1/2 x 9 inch sample prior to treatment. The sample was notched at 1 1/2 inch intervals to mark the specimen division, then each specimen was notched 1/4 inch from either side to denote the limits to be ravelled. Notching was accomplished by a razor blade while measurements were made with aluminum templates 1 x 6 1/2 inches and 1 1/2 x 6 1/2 inches respectively.

Specimens for breaking strength tests were cut from the treated samples and ravelled to the previously marked limits. In this way the same number of warp ends were broken in each individual test; in this case, the ends were 112 in number.

Testing Procedure in General.--The pH of the chemical solutions was held constant as follows: benzoic acid - pH 5.5; salicylic acid - pH 5.5; phenol - pH 6.2; ortho-phenyl phenol pH 11.2; monochlorobenzene dispersion - pH 6.2; sodium hypochlorite - pH 9.2.

The chlorobenzene was dispersed with 0.01 per cent (by volume) Triton X-100, a concentrated wetting agent manufactured by the Rhom and Haas Company, Philadelphia, Pa. All chemical agents were in aqueous solutions or dispersions.

Benzoic and salicylic acid were placed in solution by first converting them into the sodium salt with sodium hydroxide and the acid subsequently regenerated by use of dilute sulfuric acid.

Ortho-phenyl phenol was used in the form of the sodium salt since the free ortho-phenyl phenol is highly insoluble in water and immediately precipitates at a pH lower than 11.2.

A pre-arranged numbering system was used and all samples numbered with a permanent waterproof ink. All flasks were numbered in the same manner for tests being

run, in order that samples could be removed at the scheduled time. All test information was recorded in a data book for future compilation.

Tests at Room Temperature.--Solutions of the chosen concentrations were entered in 150 ml. Erlenmeyer flasks (100 ml. of solution in each) after which the sample was folded and entered. To avoid uneven chemical action, air was expelled from the folds of the cloth by agitation. The flasks were then stoppered with rubber stoppers and placed in a room where the temperature and humidity were held constant at 70°F. and 65 per cent RH. The samples were agitated once each day during the time of treatment and also checked to see that the entire sample was immersed in the solution. After remaining in the solution for the prescribed length of time, the sample was removed and prepared for testing.

Tests at 120°F and 160°F.--Solutions of the chemical agents were entered in 500 ml. porcelain beakers; the beakers were then placed in the waterbath, covered with watch glasses and allowed to reach the desired temperature. After the solution reached the temperature of the waterbath, the samples were folded and entered. The sample was weighted on three corners by 3-inch pieces of glass rod with annealed ends in order to assure even chemical action and still allow circulation.

Tests at the Boiling Point.--Tests at the boiling point of the solutions of the chemical agents were made under reflux in 500 ml. Erlenmeyer flasks. In each test 300 ml. of solution was used. The solutions were heated to the boiling point, the flasks detached from the reflux condenser, the sample entered, the flask attached again and boiling resumed for the specified length of time.

In order to keep the samples immersed and to insure uniform chemical action, three 4-inch lengths of glass rod with annealed ends were used to weight the sample down and at the same time still allow circulation of the solution through the folds of the cloth.

Sample Removal and Drying.--Solutions were poured from the beakers and flasks and the sample washed with tap water while still in the container, and later under running water. Samples treated with phenol were given a one time rinse in 70 per cent ethyl alcohol in addition to the water rinse, in order to effect complete removal of the phenol. All samples were washed until complete removal of the agent was assured. The samples were air-dried at room temperature.

Surface Examination.--Microscopic examination of longitudinal fiber sections and cross sections were made for each concentration, time and temperature range of the individual chemicals used.

Changes in Hand and Coloration.--A visual examination and comparison of the treated fabric with the untreated fabric

was made in each case to determine if any change could be detected in handle and color.

Shrinkage.--All samples were ironed at a low temperature to remove wrinkles; the sample then placed on a pressboard template 6 1/2 x 9 inches, and the amount of shrinkage measured.

Breaking Strength and Elasticity.--Dry samples were cut into individual specimens along the previously notched boundaries and then ravelled to the limits which were also previously marked.

The Scott J-2 Vertical Tester was calibrated regularly before making tests.

Prior to making any tests on the treated samples, a number of untreated samples were broken in order to obtain the average breaking strength and to determine whether the material would break in a narrow range.

The results for all tests were tabulated, averaged and the per cent loss in breaking strength calculated.

Elongation charts were made simultaneously with each breaking strength test on the Scott Tester. From these charts the elasticity was calculated.

Graphs and tables have been compiled showing the effects of some of the variables on breaking strength and are included in Chapter V and the Appendix.

CHAPTER V

DISCUSSION OF RESULTS

General.--In order to provide data for comparison with results obtained from treated samples, a total of 50 untreated samples were tested for elongation and breaking strength. The variation in the breaking strength, highest and lowest, of the untreated samples was plus or minus 4.1 per cent, while elasticity varied 1.8 per cent. Averages for the untreated samples were as follows: breaking strength 80.9 pounds; elongation 21.1 per cent. Although the variation could be expected to cause some difficulty in determining a trend, it was small enough to determine the tendency of a chemical agent to influence the strength, elasticity and shrinkage properties.

Tensile Strength.--The only strength loss shown by the fabric under test was in the 4 and 6 per cent solutions of the sodium salt of ortho-phenyl phenol at 210°F. Fabric treated at 210°F. in 4 and 6 per cent phenol gave an increase in strength of approximately 2 per cent beyond the normal variation as shown in Figure 2. In all other cases, gains or losses in strength were within the range of variation for the untreated fabric.

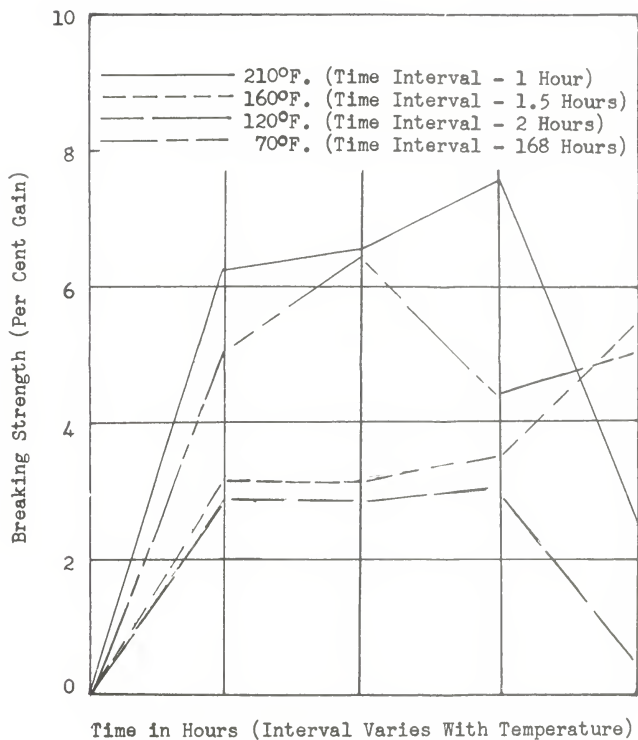


Figure 2. Effect of Time on Breaking Strength of Dacron Treated with 6 Per Cent Phenol at Various Temperatures

The action of the sodium salt of ortho-phenyl phenol is clearly a function of temperature as Dacron can withstand the action of 40 per cent sodium hydroxide at 70°F. for 10 hours with only a 2 per cent loss in strength. Figure 3 indicates variations of strength for samples treated at 210°F. in the 6 per cent concentration series; tabulated results are shown in Tables 1 through 19 in the Appendix.

Elasticity.--A gain in elasticity (per cent elongation at the point of rupture) was experienced in all cases except for samples treated with 4 and 6 per cent ortho-phenyl phenol at the boil. In both of these cases a loss in elasticity was shown. The greatest increase in elasticity was shown by phenol, monochlorobenzene and ortho-phenyl phenol. In the case of phenol the gain in elasticity was a function of time and concentration, whereas the gains experienced with monochlorobenzene and ortho-phenyl phenol were influenced to a greater degree by temperature and concentration as shown by Figures 4 through 10. Phenol shows a more consistent gain with concentration and time of treatment in all cases. Ortho-phenyl phenol gave the greatest increase in elasticity in the 4 per cent concentration series at 210°F. Benzoic acid, salicylic acid, and sodium hypochlorite gave lesser effects on elasticity; gains were in the range of a 5 per cent maximum as compared to a 20 per cent maximum for phenol.

In those cases shown graphically, it is interesting to note that the greatest gain in elasticity occurred in the first hour of treatment, thus indicating that the greatest swelling takes place in this time interval. This fact is valuable to the dyeing industry since most dye processes require maximum swelling in this period of time.

The effect of elasticity gains may be attributed to the tendency of the chemical agents to plasticize the fibers and give a molecular lubrication, thereby allowing greater elongation prior to rupture (24). Gains in elasticity are affected by the following factors: fabric geometry, shrinkage and swelling; swelling may be merely a physical change or it may be due to changes in molecular structure, thereby involving more complicated chemical changes (25, 26).

It has been proven that the effectiveness of a compound as a dyeing assistant is not solely a function of its swelling ability; however, all carriers used in this study did produce swelling to a certain degree. According to studies made in connection with the action of phenol on cellulose acetate, it is postulated that phenol is absorbed on the intermicellar fiber walls at low concentrations and as the concentration is increased the swelling pressure disrupts the internal fiber structure, opening large cavities and leading to increased water absorption and a great increase in the number of sites upon which phenol can be absorbed. The results of the investigation herein

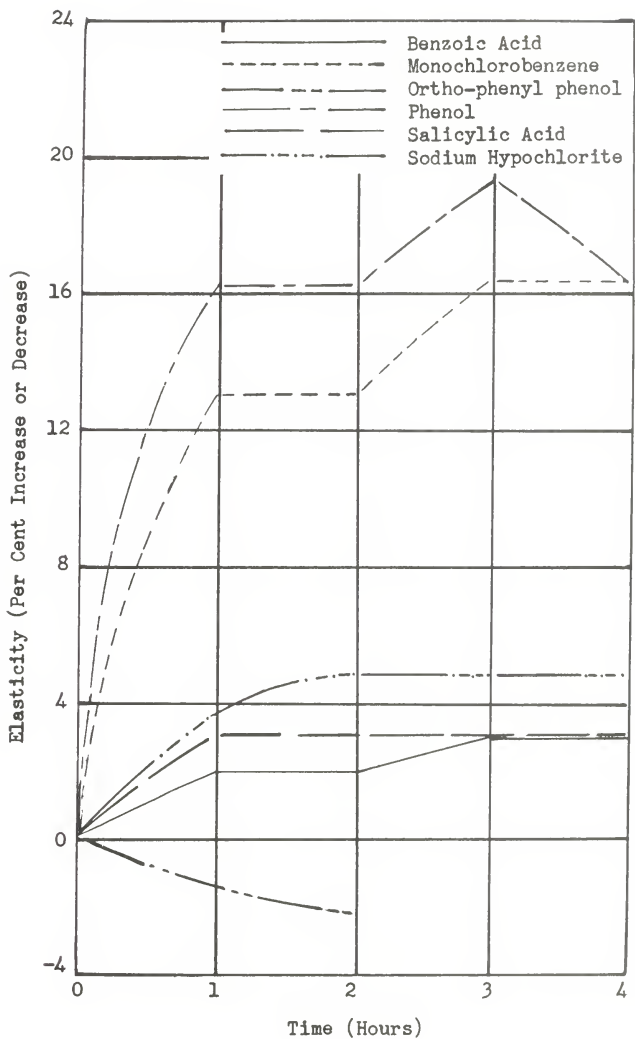


Figure 4. Effect of Time on the Elasticity of Dacron Treated in the 6 Per Cent Concentration Series at 210°F.

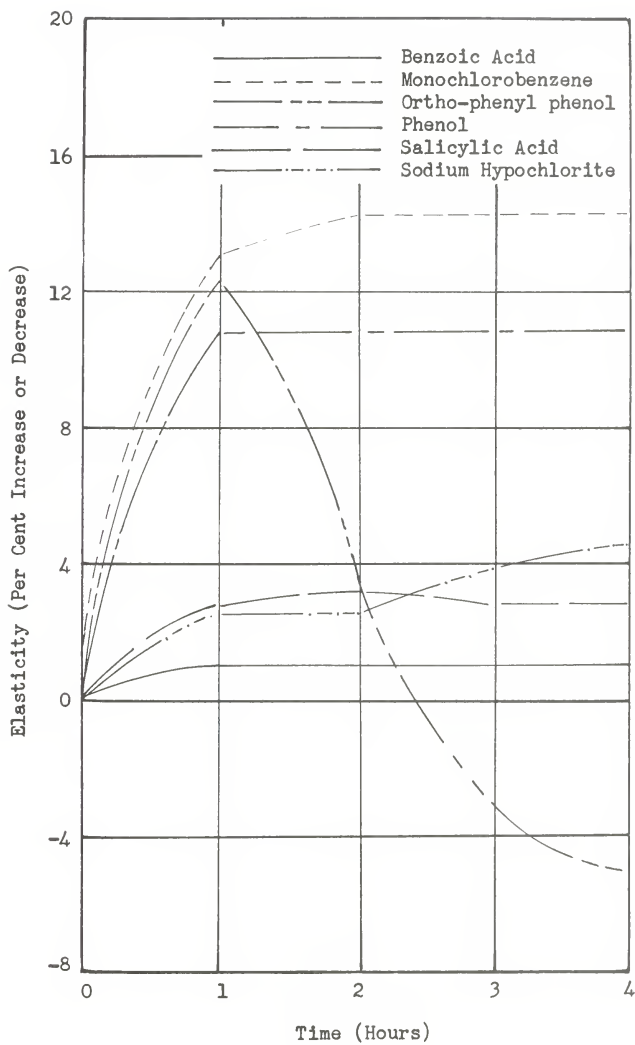


Figure 5. Effect of Time on the Elasticity of Dacron Treated in the 4 Per Cent Concentration Series at 210°F.

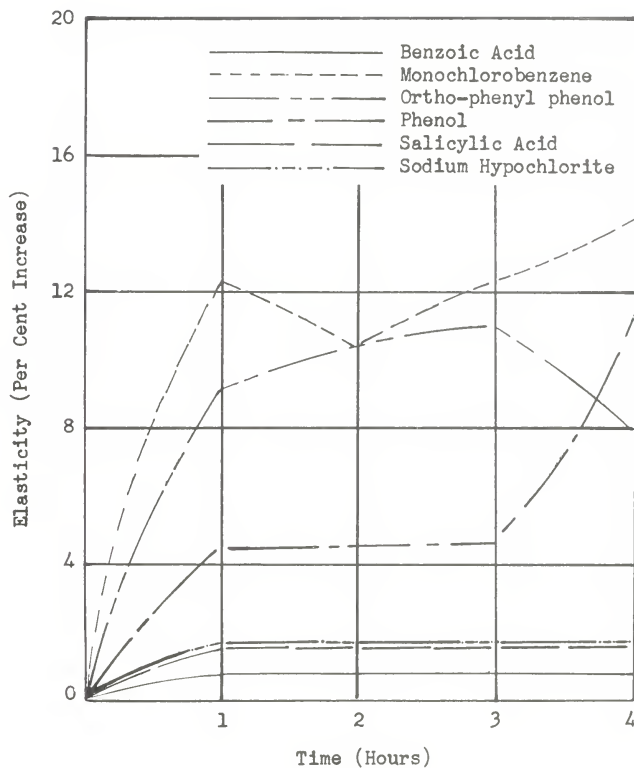


Figure 6. Effect of Time on the Elasticity of Dacron Treated in the 2 Per Cent Concentration Series at 210°F.

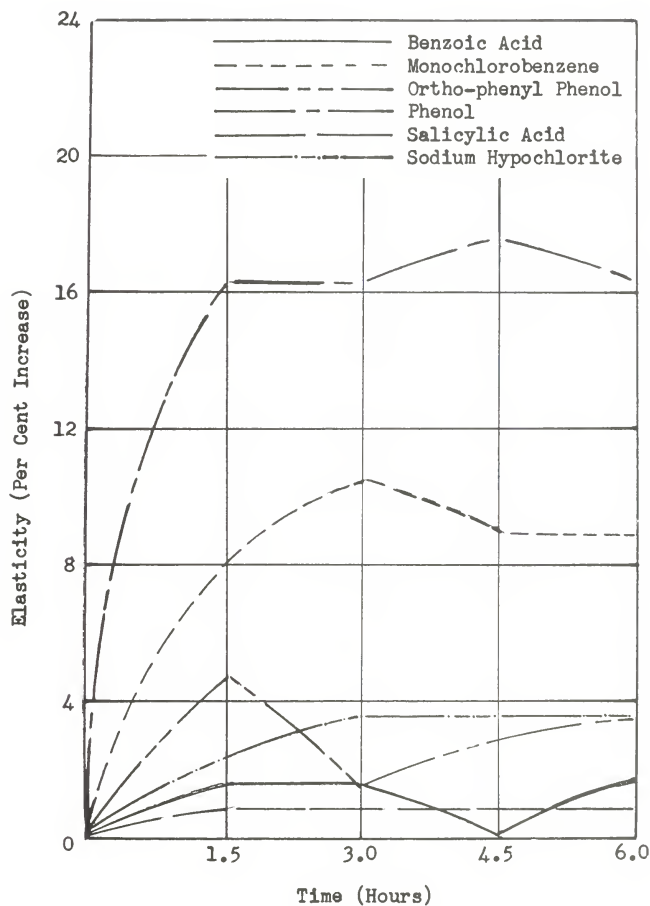


Figure 7. Effect of Time on the Elasticity of Dacron Treated in the 6 per Cent Concentration Series at 160°F.

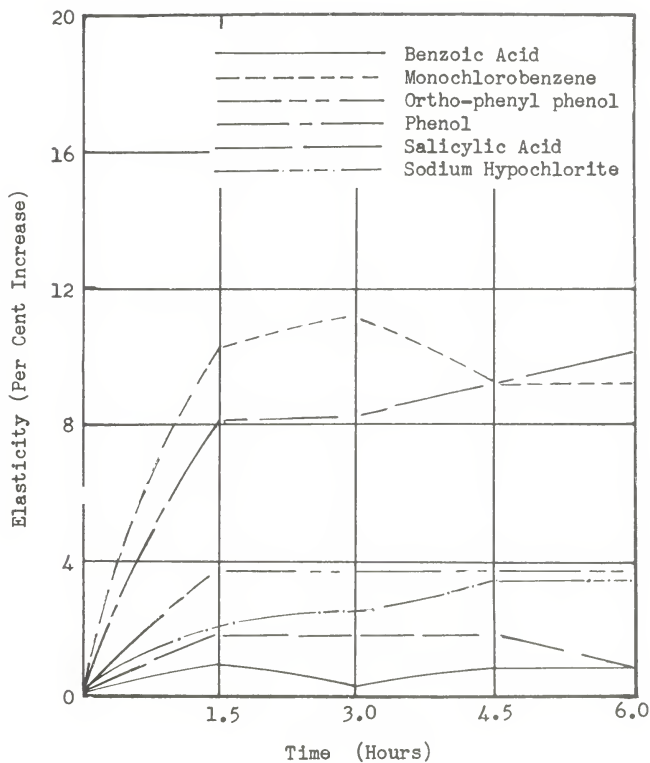


Figure 8. Effect of Time on the Elasticity of Dacron Treated in the 4 Per Cent Concentration Series at 160°F.

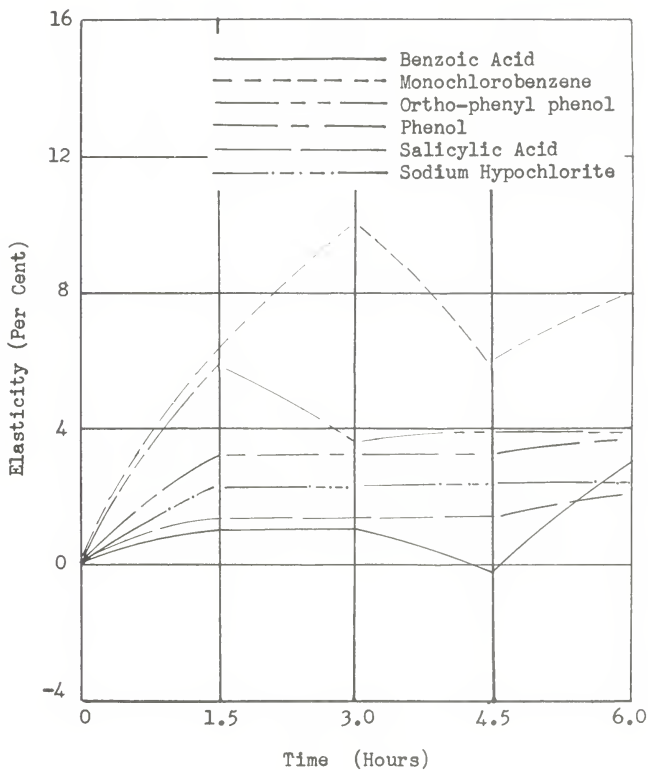


Figure 9 Effect of Time on the Elasticity of Dacron Treated in the 2 Per Cent Concentration Series at 160°F.

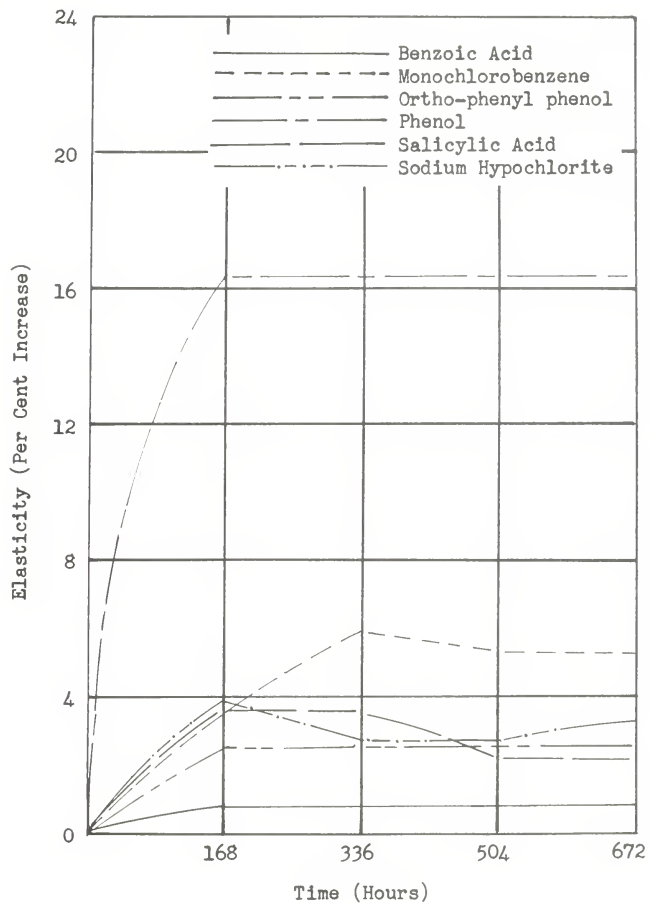


Figure 10. Effect of Time on the Elasticity of Dacron Treated in the 6 Per Cent Concentration Series at 70°F.

confirms the fact that phenol produces abnormal swelling action since the increased elasticity is a direct result of this action. In the case of phenol, it was pointed out that an ionic theory of swelling would not be applicable, as only hydrogen and phenoxide ions are in solution; these arising from dissociation of phenol. Acid solutions which contain far more hydrogen ions do not cause a swelling of the fibers and neither does sodium phenate. It is thought that the active agent must be the undissociated phenol itself and that absorption on the ester linkages must take place. It is further pointed out that chloro groups introduced into phenol increase the hydrogen bonding power and the swelling action. Although no attempt will be made to explain the chemical action of the carriers used herein, the above discussion serves to point out the fact that swelling is probably due to other than mechanical action alone (27).

Shrinkage.--With the exception of phenol, shrinkage was greatest at the higher temperatures and increased with concentration. In the case of phenol, shrinkage at 2 and 4 per cent concentrations was of the same order as above; however, at 6 per cent concentration shrinkage was dependent on time to a greater extent. This is illustrated by the fact that treatment at room temperature for 672 hours gave an increase in shrinkage of greater magnitude than at the boil for 4 hours (see Figure 17). Results of treatments at 210°F. and 160°F. are shown graphically in Figures 11 through 16, while tabulated results for all ranges of

treatment are shown in the Appendix. The phenols and monochlorobenzene gave the greatest amount of shrinkage; phenol reaching a maximum of 7.9 per cent, while benzoic acid, salicylic acid and sodium hypochlorite gave a maximum of 2.1 per cent.

In all cases the effect of fabric geometry must be considered although it is probably very slight as shrinkage in tap water was only 1 per cent for a sample treated for 4 hours at the boil. The amount of shrinkage in water would represent the contraction due to weaving strains and fabric construction. Fabric construction coupled with the swelling produced by some of the chemical agents is considered responsible for shrinkage beyond the amount noted for tap water, swelling constituting the largest factor. In general, all shrinkage was in a warpwise direction; this fact is attributed in part to the weaving process since tension on the warp was probably greater than filling tension, therefore relaxation of the strains occurred when the fabric was placed in a solution at elevated temperature. Another factor responsible for warpwise shrinkage was fabric construction (112 ends and 72 picks per inch).

Elasticity did not prove to be a function of shrinkage as might have been expected. The two did run parallel in some instances; however, in the lower temperature series where shrinkage was zero, the elasticity was above the normal variation for untreated fabric.

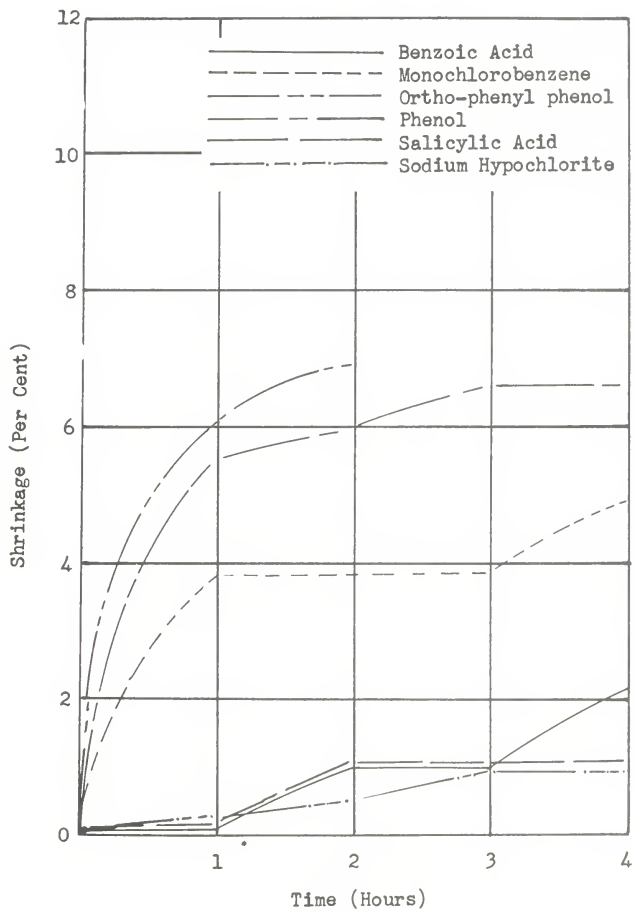


Figure 11. Effect of Time on Shrinkage of Dacron Treated in the 6 Per Cent Concentration Series at 210°F.

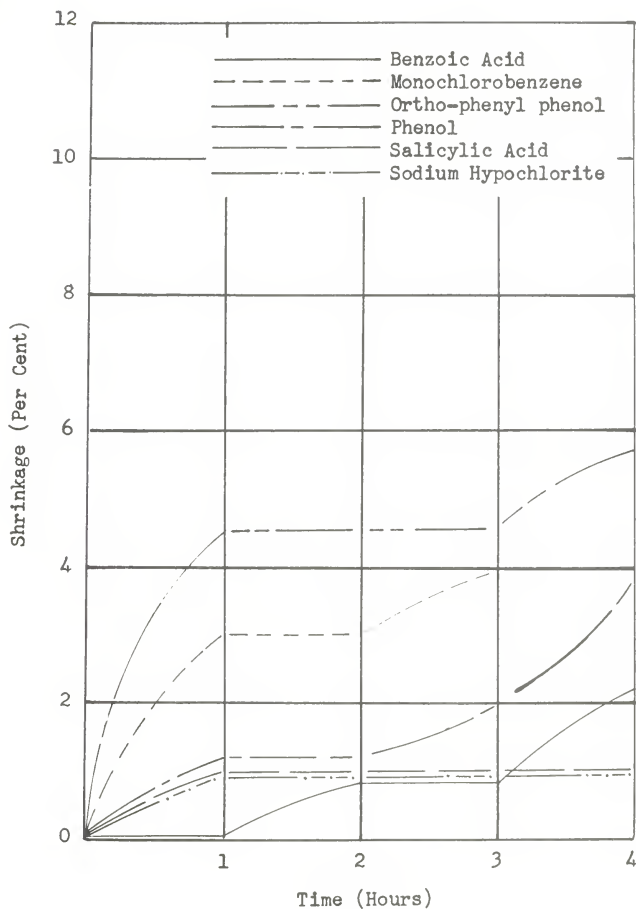


Figure 12. Effect of Time on Shrinkage of Dacron Treated in the 4 Per Cent Concentration Series at 210°F.

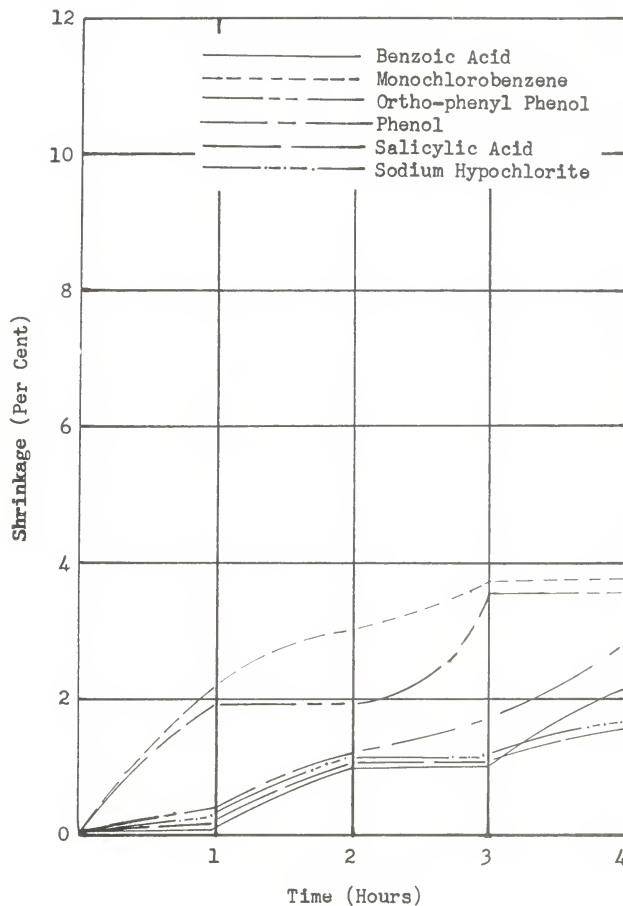
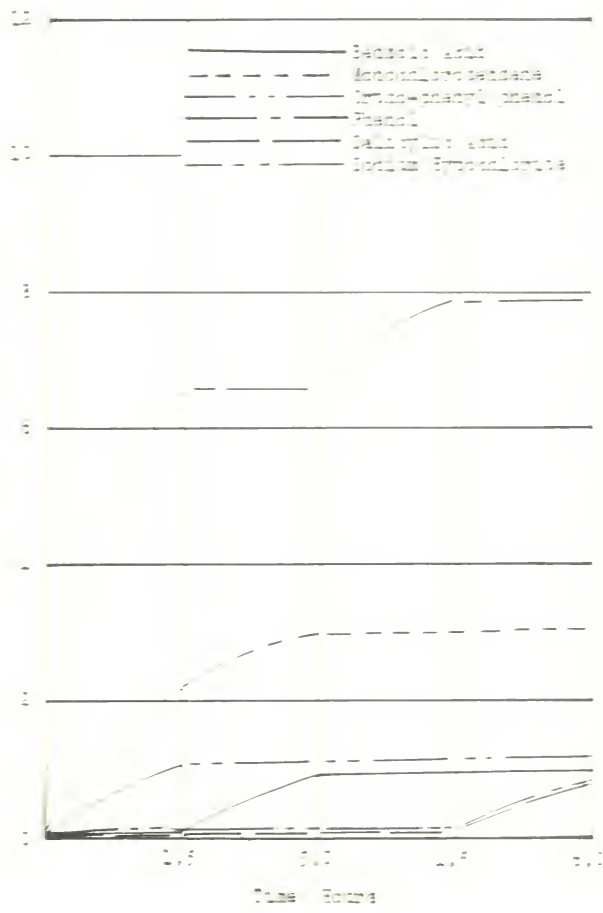


Figure 13. Effect of Time on Shrinkage of Dacron Treated in the 2 Per Cent Concentration Series at 210°F.

100
90
80
70
60

100
90
80
70
60
50
40
30
20
10
0

Shear Stress (Pascals)



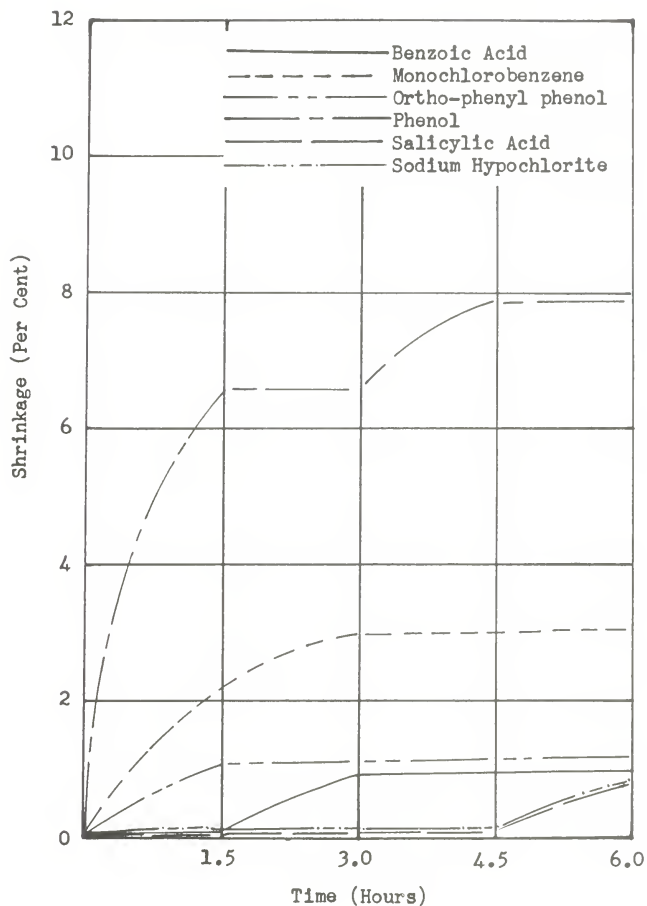


Figure 14. Effect of Time on Shrinkage of Dacron Treated in the 6 Per Cent Concentration Series at 160°F.

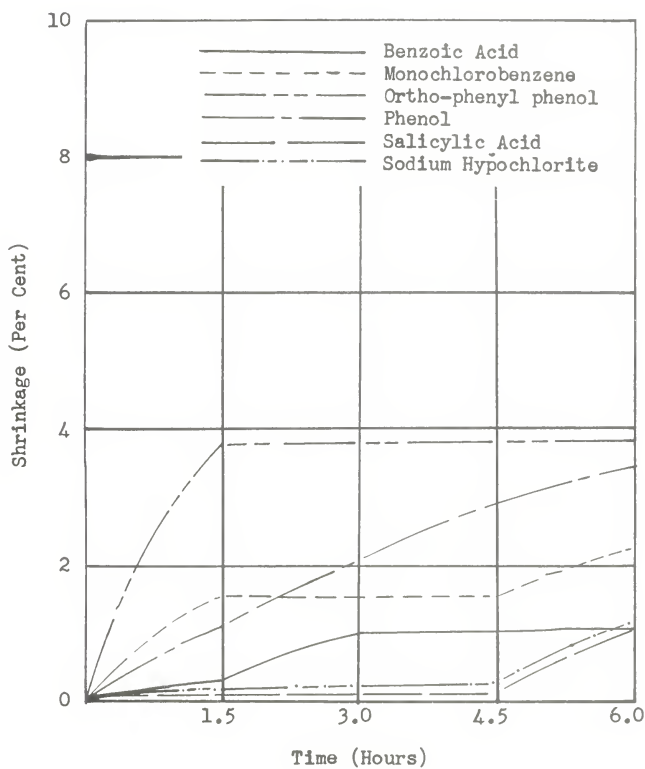


Figure 15. Effect of Time on Shrinkage of Dacron Treated in the 4 Per Cent Concentration Series at 160°F.

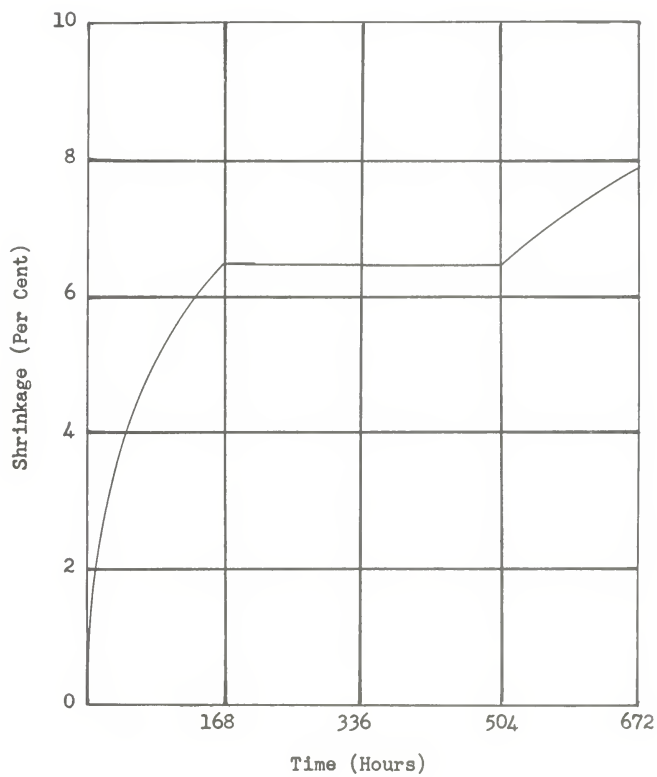


Figure 17. Effect of Time on Shrinkage of Dacron Treated with 6 Per Cent Phenol at 70°F.

Surface Characteristics.--Microscopic studies revealed no changes which could be detected. There seemed to be a slight darkening of the fiber in the case of samples treated with 6 per cent ortho-phenyl phenol; however, this may have been due to staining caused by the higher concentration of the chemical agent.

The fabric hand and appearance were unchanged for samples treated with benzoic acid, sodium hypochlorite and salicylic acid. Samples treated with 4 per cent phenol became slightly limp while those exposed to the action of 6 per cent phenol gave a very limp hand compared to the crisp resilient hand of untreated fabric. Samples treated with phenol at 160°F. and at the boil appeared slightly yellow in color.

In the case of monochlorobenzene the fabric hand became very limp for samples treated with 4 per cent concentration at 210°F. and for samples treated with 6 per cent concentration at 160°F. and at 210°F.

The fabric hand became limp for samples treated with 2, 4, and 6 per cent concentrations of ortho-phenyl phenol at 210°F. and for samples treated with 4 and 6 per cent concentrations at 160°F. Samples treated at 210°F. and 160°F. with 4 and 6 per cent concentrations gave evidence of yellowing. This discoloration was due to the agent itself.

CHAPTER VI

CONCLUSIONS

All remarks that follow, until otherwise qualified, must be applied strictly to the six chemicals employed in this study.

The tensile strength of Dacron is unaffected by benzoic acid, salicylic acid, phenol, monochlorobenzene and sodium hypochlorite. The tensile strength is affected by the sodium salt of ortho-phenyl phenol in concentrations of 4 per cent and above, at elevated temperatures.

Elasticity is a function of swelling more so than of shrinkage. Phenol and monochlorobenzene produce the greatest magnitude of swelling and possibly cause rearrangement of the molecular structure of the fiber. No attempt has been made to draw conclusions from the results of this investigation as to the nature of the chemical action of agents used or of the possible rearrangements of the fiber structure.

Those chemicals causing Dacron to exhibit the least change in breaking strength, elasticity, and shrinkage were benzoic and salicylic acid; on the basis of this study these agents would be recommended over the other carriers investigated.

It is concluded that commercial use of the agents tested would produce no deleterious effects on the physical properties of Dacron fabric, since the concentration range would be less than those used herein.

CHAPTER VII

RECOMMENDATIONS

It is known that swelling agents or carriers, of necessity, must produce some intermicellar change in the character of polyethylene terephthalate in order for dyes to penetrate the fiber structure. A study of the intermicellar space created by these agents would provide information which may lead to the nature of the action of these agents. Studies such as these may be carried out on the Dacron compliment of cellophane known as Mylar.

It seems reasonable to assume that phenolic bodies produce great swelling as evidenced in the increased elasticity; therefore, it must produce some changes in the crystalline structure either by hydrogen bonding or rupture of the bonding forces. A study of the action of these agents by X-ray diffraction methods should prove interesting and informative.

There is an extreme dearth of published information on the action of carriers or swelling agents on polyethylene terephthalate in either the drawn or undrawn state. It would seem that studies of the action of carriers on the amorphous undrawn polymer would be of value in estimating and evaluating the carrier action in the crystalline regions when the fiber is in the highly oriented state.

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APPENDIX

Table 1. Exposure in Tap Water

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	0	0.2	0.1
210	2	0.5	0.8	0.4
210	3	1	0.8	-0.4
210	4	1	0.8	0.1
160	1.5	0	0.3	0.4
160	3	0	0.8	-1.1
160	4.5	0	0.8	2.1
160	6	0.8	0.8	0.6
120	2	0	0.8	0.6
120	4	0	0.8	-1.1
120	6	0	0.8	1.7
120	8	0	0.8	-0.4
70	168	0	0.8	1.2
70	336	0	0.8	-0.9
70	504	0	0.8	1.2
70	672	0	0.8	0.1

*Average of 5 tests. Losses indicated by a minus.

Table 2. Exposure in 2 Per Cent Benzoic Acid

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	0	0.8	-0.6
210	2	1	0.8	1.9
210	3	1	0.8	-0.6
210	4	2.1	0.8	0.0
160	1.5	0	0.8	0.6
160	3	1.0	0.8	0.8
160	4.5	1	-0.3	-1.3
160	6	1	2.8	1.7
120	2	0	0.8	0.6
120	4	0	0.8	-0.6
120	6	0	0.8	-0.6
120	8	0	0.8	-0.6
70	168	0	0.8	-2.4
70	336	0	0.8	-1.1
70	504	0	0.8	-1.2
70	672	0	0.8	-1.1

*Average of 5 tests. Losses indicated by a minus.

Table 3. Exposure in 4 Per Cent Benzoic Acid

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	0	0.8	1.7
210	2	1	0.8	2.3
210	3	1	0.8	1.6
210	4	2.1	0.8	-0.6
160	1.5	0	0.8	-0.4
160	3	1	0.3	0.0
160	4.5	1	0.8	-2.3
160	6	1	0.8	0.6
120	2	0	0.8	-0.9
120	4	0	0.3	-0.9
120	6	0	0.3	-0.6
120	8	0	0.8	0.0
70	168	0	0.8	0.0
70	336	0	0.3	-1.1
70	504	0	0.8	0.6
70	672	0	0.8	-0.9

* Average of 5 tests. Losses indicated by a minus.

Table 4. Exposure in 6 Per Cent Benzoic Acid

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	0	1.8	1.6
210	2	1	1.8	1.2
210	3	1	2.8	1.7
210	4	2.1	2.8	0.6
160	1.5	0	1.8	0.0
160	3	1	1.8	0.9
160	4.5	1	0.8	-0.6
160	6	1	1.8	2.3
120	2	0	0.8	0.4
120	4	0	0.8	1.1
120	6	0	0.8	2.6
120	8	0	0.8	0.1
70	168	0	0.8	0.0
70	336	0	0.8	-0.9
70	504	0	0.8	0.1
70	672	0	0.8	1.6

*Average of 5 tests. Losses indicated by a minus.

Table 5. Exposure in 2 Per Cent Salicylic Acid

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	0	1.8	0.6
210	2	1	1.8	-0.6
210	3	1	1.8	0.0
210	4	1.4	1.8	0.4
160	1.5	0	0.8	-0.4
160	3	0	0.8	2.3
160	4.5	0	0.8	4.3
160	6	1	1.8	2.8
120	2	0	0.3	1.2
120	4	0	0.3	3.3
120	6	0	0.3	0.9
120	8	0	1.8	3.5
70	168	0	1.8	1.2
70	336	0	0.3	-2.1
70	504	0	1.8	1.2
70	672	0	1.8	-1.1

*Average of 5 tests. Losses indicated by a minus.

Table 6. Exposure in 4 Per Cent Salicylic Acid

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	0	2.8	2.6
210	2	1	3.3	3.6
210	3	1	2.8	2.1
210	4	1	2.8	1.7
160	1.5	0	1.8	0.6
160	3	0	1.8	0.6
160	4.5	0	1.8	1.7
160	6	1	0.8	6.1
120	2	0	0.8	1.1
120	4	0	0.8	3.3
120	6	0	0.8	0.9
120	8	0	0.8	-0.6
70	168	0	1.8	1.7
70	336	0	1.8	-1.1
70	504	0	1.8	-0.6
70	672	0	1.8	0.9

*Average of five tests. Losses indicated by a minus.

Table 7. Exposure in 6 Per Cent Salicylic Acid

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	0	2.8	1.2
210	2	1	2.8	2.7
210	3	1	2.8	-0.4
210	4	1	2.8	3.6
160	1.5	0	0.8	1.7
160	3	0	0.8	3.0
160	4.5	0	0.8	2.8
160	6	1	0.8	2.1
120	2	0	0.2	-1.1
120	4	0	0.8	-1.1
120	6	0	0.8	-0.4
120	8	0	0.8	-0.4
70	168	0	3.3	2.7
70	336	0	3.3	0.1
70	504	0	2.3	1.6
70	672	0	2.3	0.1

* Average of 5 tests. Losses indicated by a minus.

Table 8. Exposure in 2 Per Cent Phenol

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	0	4.9	2.6
210	2	1	4.9	3.8
210	3	1.4	4.9	6.5
210	4	2.9	11.1	3.6
160	1.5	0	2.8	1.7
160	3	1	2.8	3.0
160	4.5	1	2.8	3.8
160	6	1	3.3	4.4
120	2	0	1.3	0.4
120	4	0	1.3	2.1
120	6	0	1.3	0.1
120	8	0	1.8	2.3
70	163	0	1.8	1.1
70	336	0	1.8	3.3
70	504	0	1.8	4.7
70	672	0	1.8	4.7

*Average of 5 tests. Losses indicated by a minus.

Table 9. Exposure in 4 Per Cent Phenol

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	1	11.1	5.8
210	2	1	11.1	4.7
210	3	2.1	11.1	5.0
210	4	3.9	11.1	0.1
160	1.5	1	8.0	2.7
160	3	2.1	8.0	1.4
160	4.5	2.9	9.0	3.9
160	6	3.4	10.1	6.3
120	2	0	3.9	5.4
120	4	0	3.3	4.0
120	6	1	3.3	2.7
120	8	1	8.0	3.8
70	168	0	10.7	7.3
70	336	0	8.5	6.1
70	504	0	8.0	4.0
70	672	0	10.1	6.1

*Average of 5 tests. Losses indicated by a minus.

Table 10. Exposure in 6 Per Cent Phenol

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	5.5	16.4	6.3
210	2	6.0	16.4	6.6
210	3	6.5	17.5	7.5
210	4	6.5	16.4	2.7
160	1.5	6.5	16.4	3.3
160	3	6.5	16.4	3.3
160	4.5	7.7	17.5	3.6
160	6	7.7	18.5	5.3
120	2	5.1	14.3	3.3
120	4	5.1	15.4	3.1
120	6	5.1	16.4	3.2
120	8	6.5	16.4	0.6
70	168	6.5	16.4	5.1
70	336	6.5	16.4	6.3
70	504	6.5	16.4	4.3
70	672	7.9	16.4	5.1

* Average of 5 tests. Losses indicated by a minus.

Table 11. Exposure in 2 Per Cent Sodium Hypochlorite

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	0	1.8	0.0
210	2	1	1.8	-0.6
210	3	1	1.8	0.4
210	4	1.4	1.8	0.4
160	1.5	0	1.8	0.4
160	3	0	1.8	2.3
160	4.5	0	1.8	1.2
160	6	1	1.8	1.7
120	2	0	1.8	0.0
120	4	0	1.8	-0.4
120	6	0	1.8	-0.4
120	8	0	1.8	-1.9
70	168	0	3.9	2.6
70	336	0	1.8	-1.1
70	504	0	1.8	-1.6
70	672	0	1.8	-0.8

*Average of 5 tests. Losses indicated by a minus.

Table 12. Exposure in 4 Per Cent Sodium Hypochlorite

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	0	2.8	-1.9
210	2	1	2.8	-1.6
210	3	1	3.9	-0.9
210	4	1	4.9	0.6
160	1.5	0	1.8	1.7
160	3	0	2.3	0.6
160	4.5	0	2.8	1.1
160	6	1	2.8	0.0
120	2	0	1.8	-1.3
120	4	0	1.8	-0.4
120	6	0	1.8	-0.7
120	8	0	1.8	-0.6
70	168	0	1.8	-0.6
70	336	0	1.8	0.3
70	504	0	1.8	0.6
70	672	0	1.8	0.8

* Average of 5 tests. Losses indicated by a minus.

Table 13. Exposure in 6 Per Cent Sodium Hypochlorite

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	0	3.9	2.3
210	2	1	4.9	-1.6
210	3	1	4.9	0.0
210	4	1	4.9	-2.1
160	1.5	0	2.4	0.4
160	3	0	3.9	2.1
160	4.5	0	3.9	1.1
160	6	1	3.8	0.0
120	2	0	0.8	0.4
120	4	0	0.8	0.6
120	6	0	0.8	0.0
120	8	0	0.8	-0.9
70	168	0	3.9	1.6
70	336	0	2.8	0.9
70	504	0	2.8	0.1
70	672	0	3.3	1.1

*Average of 5 tests. Losses indicated by a minus.

Table 14. Exposure in 2 Per Cent Monochlorobenzene

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	2.1	12.2	0.6
210	2	2.9	10.1	0.0
210	3	3.9	12.2	2.3
210	4	3.9	14.3	-1.3
160	1.5	1	6	0.6
160	3	1	10.1	2.3
160	4.5	1	6.0	1.2
160	6	2.1	8.0	-1.3
120	2	0	2.8	1.2
120	4	0	3.3	1.7
120	6	0	3.3	1.7
120	8	0	3.3	0.0
70	168	0	2.8	-0.4
70	336	0	3.3	1.1
70	504	0	3.3	1.1
70	672	0	3.9	1.2

* Average of 5 tests. Losses indicated by a minus.

Table 15. Exposure in 4 Per Cent Monochlorobenzene

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	2.9	12.2	-0.4
210	2	2.9	13.8	0.4
210	3	2.9	13.8	1.6
210	4	3.9	13.8	-4.8
160	1.5	1.4	10.1	0.6
160	3	1.4	10.7	1.1
160	4.5	1.4	9.0	-0.9
160	6	2.1	9.0	-1.3
120	2	0	2.8	1.1
120	4	0	2.8	1.1
120	6	0	3.9	1.1
120	8	0	3.9	0.6
70	168	0	3.9	1.2
70	336	0	3.9	0.4
70	504	0	3.9	-1.6
70	672	0	3.9	2.6

*Average of five tests. Losses indicated by a minus.

Table 16. Exposure in 6 Per Cent Monochlorobenzene

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	3.9	13.3	0.6
210	2	3.9	13.3	0.4
210	3	3.9	16.4	-1.3
210	4	4.8	16.4	-2.3
160	1.5	2.1	8.0	2.1
160	3	2.9	10.1	2.1
160	4.5	2.9	9.0	0.1
160	6	2.9	9.0	0.0
120	2	0	2.8	0.4
120	4	0	2.8	0.0
120	6	0	3.3	0.0
120	8	0	3.3	2.1
70	168	0	2.8	-0.9
70	336	0	5.5	3.6
70	504	0	4.9	-0.4
70	672	0	4.9	0.0

*Average of 5 tests. Losses indicated by a minus.

Table 17. Exposure in 2 Per Cent Ortho-Phenyl Phenol
(Sodium Salt)

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	2.1	9	5.7
210	2	2.1	10.1	3.3
210	3	3.9	11.1	4.0
210	4	3.9	8.0	2.3
160	1.5	0	6.0	2.8
160	3	0	3.3	2.1
160	4.5	1	3.9	1.6
160	6	1.4	3.9	1.1
120	2	0	3.9	2.8
120	4	0	3.9	2.8
120	6	0	3.9	2.8
120	8	0	3.9	3.8
70	168	0	2.8	2.6
70	336	0	2.8	-0.3
70	504	0	0.8	0.0
70	672	0	2.4	1.6

*Average of 5 tests. Losses indicated by a minus.

Table 18. Exposure in 4 Per Cent Ortho-Phenyl Phenol
(Sodium Salt)

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	4.8	12.2	-6.0
210	2	4.8	3.9	-8.0
210	3	4.8	-2.9	-47.5
210	4	5.5	-4.9	-67.8
160	1.5	0	3.9	2.8
160	3	0	3.9	1.7
160	4.5	1.4	3.9	0.9
160	6	1.4	3.9	0.9
120	2	0	4.9	2.2
120	4	0	4.1	4.0
120	6	0	4.9	2.1
120	8	0	4.9	1.7
70	168	0	2.3	0.1
70	336	0	2.3	1.1
70	504	0	2.8	0.6
70	672	0	3.9	1.2

*Average of 5 tests. Losses indicated by a minus.

Table 19. Exposure in 6 Per Cent Ortho-Phenyl Phenol
(Sodium Salt)

Temperature (°F.)	Time (Hrs.)	Shrinkage (Per cent)	Increase or Decrease in Elasticity* (Per cent)	Loss or Gain in Strength* (Per cent)
210	1	6.2	-1.3	-40.5
210	2	6.8	-2.4	-61.5
210	3	(Sample Degraded)**		
210	4	(Sample Degraded)**		
160	1.5	1	5.5	3.3
160	3	1	1.8	-2.1
160	4.5	1	2.8	1.2
160	6	1	3.9	-5.6
120	2	0	2.8	0.6
120	4	0	4.9	4.0
120	6	0	3.9	1.9
120	8	0	2.8	2.6
70	168	0	2.8	-0.9
70	336	0	2.8	-1.3
70	504	0	2.8	-1.9
70	672	0	2.8	0.1

*Average of 5 tests. Losses indicated by a minus.

**Sample was degraded to such an extent that tests could not be performed.

Thesis
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